INDENTATION FRACTURE TOUGHNESS OF Y-TZP DENTAL CERAMICS

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Abstract: The purpose of this study was to investigate and analyze fracture toughness (K_{Ic}) of yttria stabilized tetragonal zirconia (Y-TZP) dental ceramics by the Vickers indentation fracture test (VIF). In order to determine fracture toughness, the Vickers indenter was used under the load of 294.20 N (*HV*30). The cracks, which occur from the corners of a Vickers indentation, were measured and used for fracture toughness determination, through the different mathematical models (according to Anstis, Casellas, Palmqvist and Niihara).

Keywords: Y-TZP ceramics, Indentation test, Fracture toughness

1. Introduction

The application of ceramics as structural materials is based on their light weight combined with their high temperature resistance, high hardness and stiffness (modulus of elasticity), chemical inertness and elevated wear resistance. However, the fracture toughness of ceramics is still poor, compared to that of metals and composites.

Zirconia ceramics have received considerable attention along the last decades because of the feasibility to obtain relatively high fracture toughness values through induced microstructural and phase assemblage changes [1]. This possibility is related to the stress-induced phase transformation that tetragonal zirconia may undergo to monoclinic (t $\rightarrow$ m). Such phase transformation involves a volumetric increase (~4%) that may induce compressive stresses in the crack area, preventing its propagation, thus increasing the fracture toughness [2]. This strengthening mechanism is known as the transformation toughening and makes ZrO_2 much tougher in comparison with all other ceramic materials [1, 3, 4, 5-8].

The fracture resistance of brittle materials, such as ceramics, is generally characterized by the fracture toughness value [9-11]. The fracture of the brittle ceramics is usually controlled by the mode I fracture toughness. Simple dimensional analysis of a body containing the crack with the length of $2a$, subjected to the stress σ, shows that the stress intensification at the crack tip is:

$$K_I = \sigma \cdot Y \cdot a^{1/2} \tag{1}$$

where K_I represents the stress intensity factor and Y is a dimensionless constant that depends on sample geometry and crack configuration. The concept of K_I is derived from the linear elastic fracture mechanics, LEFM. It represents the magnitude of the stress field in the crack tip region. The subscript I refers to the tensile or opening mode condition, as opposed to a shear mode, specified with indices II or III. Thus the variable known as the fracture toughness, K_{Ic}, refers to the critical value of the stress intensity factor in the opening mode when the fracture initiates and the unstable crack propagation occurs.

Scientists have developed numerous test methods of measuring fracture toughness [12-16]. The Vickers indentation fracture test, or VIF was adopted as popular experimental technique for the determination of the fracture toughness of all brittle materials, including ceramics [12, 13, 17 – 20]. The method consists of measuring the total length of cracks

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emanating from the four corners of a Vickers indentation as a consequence of applied load. The crack lengths are in an inverse proportion to the toughness of the material. By the measurement of the crack lengths, it is possible to estimate K_{Ic}. There is a large number of equation modifications, more than 30, by different scientists in order to satisfy required value of fracture toughness. Scientists have been using different equations for Palmqvist and radial-median cracks or for both type of cracks at the same time what leads to different results for the same material and test conditions. Therefore, it is necessary to identify the crack profile (Fig. 1) and to select the appropriate equation (model) for the calculation of the exact fracture toughness value.

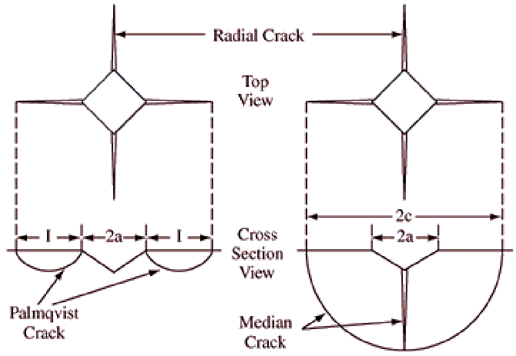

Fig. 1. Crack formation by Vickers indentation [18].

The goal of this work was to investigate the crack type and evaluate the fracture toughness (K_{Ic}) of yttria-stabilized zirconia (Y-TZP) dental ceramics by the Vickers indentation fracture toughness (VIF) test.

2. Experiment

All experiments were performed on Y-TZP dental ceramics with 4.1 wt. % Y_2O_3. Samples were provided by BruxZir, Glidewell Laboratories (Newport Beach, CA, USA). The manufacturer provided samples in the shape of square plates, 10 × 10 × 2 mm, sintered by the usual sintering regime for production of ceramic restorations at the Glidewell Laboratories. The chemical composition of the investigated Y-TZP dental ceramics, according to the manufacturer's declaration, is shown in Table 1.

Table 1. Chemical composition of the Y-TZP dental ceramics expressed as weight percent (wt. %).

Sample	wt. %						
	Y_2O_3	HfO_2	Al_2O_3	SiO_2	Fe_2O_3	Na_2O	ZrO_2
Y-TZP ceramics	4.1	4.0	0.34	<0.01	<0.01	<0.01	Balance

Fracture toughness determination by the VIF technique, as well as measuring the Vickers hardness, proceeds by first preparing a high quality polished test specimen surface. Surface preparation prior to hardness measurements (grinding and polishing) affects the hardness and fracture toughness values [21-23]. Therefore, it is important to adequately prepare sample surface according to standard ceramographic procedure. Prior to examination, the samples were polished with a series of diamond pastes up to 1 μm finish.

The polished specimen surface is then penetrated by a Vickers pyramidal indenter to create a deformed region beneath and in the vicinity of the indentation which generate the cracks emanating from the corners of the square Vickers indent. Fracture toughness determination and hardness measurement were performed under the load 294.20 N (*HV*30). Thirty indentations were made under ambient laboratory conditions on the referent hardness tester Indentec, UK; type: 5030 TKV, while the diagonal and crack were measured on an optical microscope (Olympus Imaging Corp., Tokyo, Japan), immediately after unloading.

Vickers indentation with characteristic values measured after removal of load is presented in Fig. 2.

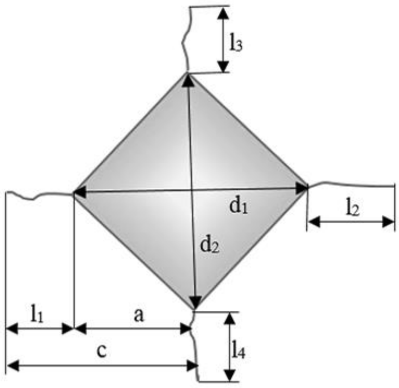

Fig. 2. Vickers indentation with characteristic values.

The length of cracks, the indentation load, the diagonal of indentation, the hardness and elastic modulus of the material and an empirical calibration constant are used to calculate fracture toughness by different equations according to Anstis [12], Casellas [7, 24], Palmqvist [25] and Niihara [18, 22]. The models (equations) are presented in Table 2.

Table 2. Fracture toughness models (F – applied load; c – crack length from the center of the impression to the crack tip; E – Young's modulus; HV – Vickers hardness; T – total crack length, $T=l_1+l_2+l_3+l_4$; a – half value of the indentation diagonal).

Model	Equation
Antis	$K_{Ic} = 0.016 \cdot \frac{F}{c^{3/2}} \cdot \left(\frac{E}{HV}\right)^{1/2}$ [MPa·m$^{1/2}$]
Casellas	$K_{Ic} = 0.024 \cdot \frac{F}{c^{3/2}} \cdot \left(\frac{E}{HV}\right)^{1/2}$ [MPa·m$^{1/2}$]
Palmqvist	$K_{Ic} = 0.0028 \cdot HV^{1/2} \cdot \left(\frac{F}{T}\right)^{1/2}$ [MPa·m$^{1/2}$]
Niihara	$K_{Ic} = 0.000165 \cdot E^{0.4} \cdot F^{0.6} \cdot a^{-0.7} \cdot \left(\frac{c}{a}\right)^{-1.5}$ [MPa·m$^{1/2}$]

3. Results

Statistical analysis results of Vickers hardness (HV) obtained from thirty measurements, including the minimum, the maximum, the arithmetic average value and its standard deviation are summarized in the Table 3.

Table 3. Statistical analysis results for the measured hardness (HV) of Y-TZP dental ceramics.

Hardness	Average	Standard deviation	Minimum	Maximum
*HV*30	1337	10	1313	1355

Vickers indentations before polishing are presented in Fig. 3a) and 3b). As can be seen from the figures on the corners of indentations cracks occur as a consequence of applied load. Still, there is no extensive lateral cracking or spalling around the indentations. The surrounding area of Vickers indentation is free of multiple cracking and the crack length could be easily measured.

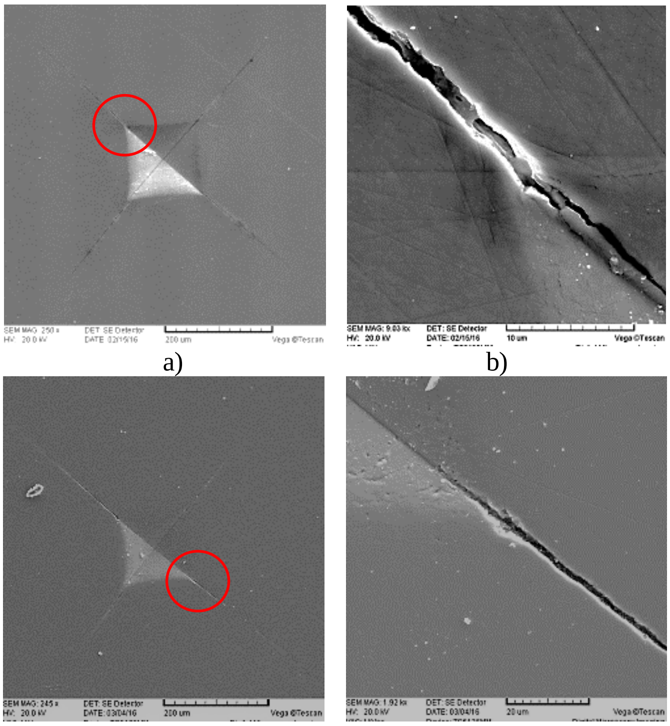

Fig. 3. The SEM micrograph of the Vickers indentation and cracks at the test load 294.20 N: a) before polishing; b) enlarged section from a); c) after polishing the surface layers; d) enlarged section from c).

Table 4 shows the indentation diagonal length and crack length measured at the load 294.20 N.

Table 4. Statistical analysis of the diagonal half length (*a*, mean value ± standard deviation), and crack length (*c*, mean value ± standard deviation) of Y-TZP dental ceramics.

F, N	*a*, μm	*c*, μm	*c*/*a*
294.20	102.0±0.4	282.8±5.6	2.77

To define the crack system, indented sample was polished repeatedly, using 6 μm diamond as an abrasive. At the load of 294.20 N the cracks emanating from the indentation corners were connected as it is clearly discerned in Fig. 3c) and 3d).

The cracks that originated from the corners of the Vickers indentation were used to compute the fracture toughness by the VIF method. For the determination of fracture toughness by the VIF method were used equation proposed by Antis, Casellas, Palmqvist and Niihara. For the Young's modulus *E*, a value of 210 GPa was assumed [21]. The results of VIF measurements are summarized in Table 5.

Table 5. Statistical analysis of the fracture toughness (K_{Ic}) of Y-TZP dental ceramics.

Model	Average $MPa \cdot m^{1/2}$	Standard deviation $MPa \cdot m^{1/2}$	Minimum $MPa \cdot m^{1/2}$	Maximum $MPa \cdot m^{1/2}$
Palmqvist	6.47	0.11	6.28	6.77
Antis	3.96	0.12	3.74	4.26
Casellas	5.95	0.18	5.61	6.40
Niihara	5.72	0.17	5.39	6.16

4. Discussion

The major difficulties in determining reliable fracture toughness values by VIF technique are: defining the type of formed cracks, precise measurements of the crack length, as well as an application of a suitable crack model equation.

Two types of crack system are produced by a Vickers indenter, i.e., the Palmqvist crack system with half-ellipse sub-structure and radial-median or half-penny crack system [26, 27]. Generally, the high toughness material shows Palmqvist crack system. However, most materials will present both crack systems dependent on the amount of load [26, 27]. For brittle materials the radial-median cracks were formed in the high-load regime [22]. There are two ways to distinguish the crack system present in the analysed material. One way to define the crack system consists on verification of c/a ratio (c is the crack length from the centre of the indent to the crack tip and a is the half value of the indentation diagonal, Fig. 2, Table 4). If it is larger than 2.5, than the material shows radial-median crack system [21-23, 27]. The other way to differentiate between the two types of crack system is to polish the surface layers away. The Palmqvist cracks will detach from the indentation, while the radial-median crack system will always remain connected to the inverted pyramid of the indent [26, 27], as shown in Fig. 3c) and 3d). According the both approaches, at the indentation load 294.20 N, radial-median crack system were observed, i.e. ratio of c/a was higher than 2.5 (Table 4) and after polishing cracks were connected to the inverted pyramid of the indent.

When the different equations are applied to estimate the fracture resistance then the fracture toughness varies for the same material [17]. This was shown by the application of the Anstis, Casellas, Palmqvist and Niihara models, which consider Young's modulus, applied indentation load, indentation size, as well as crack size.

Despite the Anstis equation be widely applied for estimates the fracture toughness, its results are not applicable for investigated ceramics. It can be attributed to the fact that the equation proposed by Anstis with the calibration constant 0.016 underestimates the residual stress field. Some authors, as Casellas, have proposed correction for this parameter (more than 20%) to be used in ceramics containing zirconia.

5. Conclusions

The results presented in this paper can be summarized as follows:

- Advantages of the Vickers indentation fracture test (VIF) are that it requires only a small volume of material, minimal specimen preparation, low costs and simple experimental procedure.
- The fracture toughness values of Y-TZP dental ceramics depend on the chosen model of the fracture resistance. The highest fracture toughness value was obtained by Palmqvist model while the lowest value was measured by Antis. Therefore, it is important to choose the appropriate model, because the differences in the measured fracture toughness can be very high. The most applicable models for the analysis of the fracture toughness of the Y-TZP dental ceramics are Palmqvist, Casellas and Niihara, but only when high indentation loads are applied.
- Y-TZP dental ceramics has shown the radial-median crack system which is in the agreement with the obtained values of c/a ratio, which is higher than 2.5 and indentation load of 294.20 N causes the merging of the cracks with the corners of the indentation.

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